

PATENT ATTORNEY DOCKET NO. 47113-0451

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	
Bjorn LUNGBERG)	Confirmation No.: 3331
Application No.: 10/807,376))	Group Art Unit: 1775
Filed: March 24, 2004)	Examiner: Archene A. Turner
For: OXIDE COATED CUTTING TOOL)	
Commissioner for Patents Arlington, VA 22202		
Sir		

SUBMISSION OF PRIORITY DOCUMENT

Under the provisions of 35 U.S.C. § 119, Applicant hereby claims the benefit of the filing date of Swedish Application No. 0300930-5, filed April 1, 2003 for the above-identified United States Patent Application.

In support of Applicant's claim for priority, filed herewith is one certified copy of the above.

Respectfully submitted,

DRINKER, BIDDLE & REATH LLP

By:

Dated: October 30, 2007

Customer No. 55694

DRINKER BIDDLE & REATH LLP 1500 K. Street, N.W., Suite 1100 Washington, DC 20005-1209

Tel: (202) 842-8800 Fax: (202) 204-0289

DC\618694\1





Intyg Certificate

Härmed intygas att bifogade kopior överensstämmer med de handlingar som ursprungligen ingivits till Patent- och registreringsverket i nedannämnda ansökan.

This is to certify that the annexed is a true copy of the documents as originally filed with the Patent- and Registration Office in a connection with the following patent application.

(71) Sökande: Sandvik Intellectual Property AB, Sandviken, SE Applicant (s)

(21) Patentansökningsnummer 0300930-5 Patent application number

(86) Ingivningsdatum 2003-04-01
Date of filing

Stockholm, 2007-10-22

För Patent- och registreringsverket For the Patent- and Registration Office

Improgend Kauliss

Avgift

Fee 170:-

OXIDE COATED CUTTING TOOL

The present invention relates to a CVD coating process for depositing α -Al₂O₃ layers at low temperatures as well as to a coated cutting tool for chipforming machining. The coated cutting tool includes at least one Al₂O₃-layer deposited according to the invented process. The coated tool shows improved toughness behaviour when used in interrupted cutting operations and improved wear resistance if the Al₂O₃ layer is deposited onto a PVD-precoated tool.

10

15

20

25

Cemented carbide cutting tools coated with various types of hard layers like TiC, TiCN, TiN and Al_2O_3 have been commercially available for years. Such tool coatings are generally built up by several hard layers in a multilayer structure. The sequence and the thickness of the individual layers are carefully chosen to suit different cutting applications and work-piece materials e g cast iron and stainless steel.

Tool coatings are most frequently deposited by Chemical Vapour Deposition (CVD) or Physical Vapour Deposition (PVD) techniques. In some rare cases also Plasma Assisted Chemical Vapour Deposition (PACVD) has been used. The CVD technique employed for coating cemented carbide tools is conducted at a rather high temperature, about 880-1000 °C. Due to this high deposition temperature and to a mismatch in thermal expansion coefficient between the deposited coating materials and the cemented carbide tool, CVD produces coatings with cooling cracks and tensile stresses. The PVD technique runs at a significantly lower temperature about 450-700 °C and it is performed under ion bombardment leading to high compressive stresses in the coating and no cooling cracks. Because of these process differences CVD-coated tools are more brittle and thereby possess inferior toughness behaviour compared to PVD coated tools.

With the CVD-technique it is possible to deposit many hard and wear resistant coating materials like Al_2O_3 , TiC, Ti(C,N), TiN $TiC_xN_yO_z$ and ZrO_2 . The microstructure and thereby the properties of these coatings can be altered quite considerably by varying the deposition conditions. If the standard CVD deposition temperature could be decreased significantly an increased toughness of the coated tool would be expected.

A noticeable improvement in performance of CVD-coated tools came about when the MTCVD (Moderate Temperature CVD)-technique begun to come into the tool industry about 5-10 years ago. An improvement in the toughness behaviour of the tool was obtained. Today the majority of tool producers use this technique. Unfortunately the MTCVD-technique is limited only to fabrication of Ti(C,N)-layers. The deposition process here takes place at temperatures in the range 700-900 °C. It uses a gas mixture of CH₃CN, TiCl₄ and H₂.

It is generally accepted that modern tool coatings also should include at least one layer of $\mathrm{Al}_2\mathrm{O}_3$ in order to achieve high crater wear resistance. Hence, it would be desirable if also high quality $\mathrm{Al}_2\mathrm{O}_3$ layers could be deposited by a CVD-process at a temperature in the range similar to that of the MTCVD TiCN-process and closer to the PVD-process temperatures if combined PVD-CVD coatings are desired.

10

15

20

25

30

40

It is well known that Al_2O_3 crystallises in several different phases: α , κ , γ , δ , θ etc. The most common CVD deposition temperature for Al_2O_3 is in the range 980-1050 °C. At these temperatures both singlephase κ - Al_2O_3 and singlephase α - Al_2O_3 can be produced or mixtures thereof. Occasionally also the θ -phase can be present in smaller amounts.

In US 5,674,564 is disclosed a method of growing a fine-grained K-alumina layer by employing a low deposition temperature and a high concentration of a sulphur compound.

In US 5,487,625 a method is disclosed for obtaining a fine grained, (012)-textured α -Al₂O₃ layer consisting of columnar grains with a small cross section (about 1 μ m).

In US 5,766,782 a method is disclosed for obtaining a fine-grained (104)-textured α -Al₂O₃ layer.

Nanocrystalline α -Al $_2$ O $_3$ layers can be deposited by PVD- and PACVD technique at low temperatures as disclosed in US 5,698,314, US 6,139,921 and US 5,516,588. However these techniques are much more technically complicated, process sensitive and have less throwing power than the CVD-technique when used for depositing α -Al $_2$ O $_3$

The κ -Al₂O₃-, γ -Al₂O₃- and α -Al₂O₃-layers have slightly different wear properties when cutting different materials. Broadly speaking the α -phase is preferred when cutting cast iron while the κ -phase is more often used when cutting low carbon steels.

Desirable is also to have means to produce $\alpha-Al_2O_3$ -layers at temperatures e g <700 °C that e g can be combined with MTCVD Ti(C,N)-layers or even can be deposited onto PVD-coated layers. Low temperature processes for $\kappa-Al_2O_3$ and $\gamma-Al_2O_3$ are disclosed in US 5,674,564 and in EP-A-1122334. Deposition temperatures in the ranges of 800-950 °C and 700-900 °C are disclosed.

In DE-A-101 15 390 a coating is disclosed consisting of a PVD-coated innerlayer with a top layer of Al₂O₃ deposited by the CVD-technique at a medium temperature. The Al₂O₃-layer can be essentially any of the modifications: κ , α , δ and amorphous. A temperature range of 700-850 °C is claimed for the deposition process. However, no method for depositing the α -Al₂O₃ phase at temperatures less than 850 °C is disclosed.

Since the α -Al₂O₃ is the high temperature stable aluminium oxide phase one would not expect it to be formed at temperatures <800 °C. EP-A-1122334 and US 5,674,564 point toward the reasonable assumption that only the metastable phases are possible to be obtained at these low temperatures. So far there have not been any reports on a CVD-process capable of depositing well crystalline α -Al₂O₃ at temperatures <800 °C that can be used as a tool coating. However, low temperature Al₂O₃ CVD-processes using Al-metallo-organic compounds have been reported e g in US 3,838,392. Such coatings are generally impure and possess no or low crystallinity and hence are not suitable as tool coatings.

The life time and the performance of a coated cutting tool are closely related to the method by which the coating is produced. As mentioned above high temperature deposition processes generally give cutting tools with lower toughness behaviour compared to coatings deposited at lower temperatures. This is due to many factors like differences in the number of cooling cracks formed in the coating, differences in the tensile stress state, influence of the process on the cemented carbide tool body e g degree of decarburisation and degree of diffusion of elements from the cemented carbide into the coating.

On the other hand high temperature deposition processes generally give better coating adhesion due to a substantial interdiffusion of materials from the tool body into the growing coating.

However, there are many cutting operations where high toughness of the tool is more important than high coating adhesion.

30

40

10

15

20

25

In such cutting operations the tougher PVD coated tools are frequently used.

PVD-coated tools generally lack wear resistance in comparison to CVD-coated tools. If the temperature of the CVD-process could be lowered for all, or at least for the majority of the coating steps then a higher toughness would be expected and such a CVD-coated tool may better complement the pure PVD-tools in operations where both toughness and high wear resistance is required.

It is an object of the present invention to provide a CVD process for depositing an α -Al₂O₃ layer at a temperature below 800 °C.

10

15

20

It is a further object of the invention to provide onto a hard tool body a wear resistant coating comprising at least one layer essentially consisting of $\alpha\text{-Al}_2\text{O}_3$ deposited by CVD at a temperature (T) below 800 °C. Other layers in the coating can be deposited by MTCVD or by PVD-technique and PACVD (plasma assisted CVD) at low temperatures.

It is still a further object of the invention to provide an alumina coated cutting tool insert, a solid carbide drill or carbide end-mill with improved cutting performance in steel.

Fig. 1-3 show Scanning Electron Microscope (SEM) micrographs in top view projections of an α -Al₂O₃ layer deposited according to the present invention in x10000 magnification. Fig 1 shows an α -Al₂O₃-layer deposited onto a PVD TiN precoated tool at 690 °C, Fig 2 onto a CVD Ti(C,N) precoated tool with a Ti(C,O) intermediate layer at 690 °C and Fig 3 onto a CVD Ti(C,N) precoated tool with a Ti(C,O) intermediate layer at 625 °C.

Fig 4 shows an XRD-diffraction pattern of a coating comprising a layer deposited by the invented low temperature Al₂O₃-process.

Surprisingly it was found after carrying out a lot of deposition experiments that also well-crystalline layers consisting of 100 % α -Al₂O₃ in fact can be deposited at such low temperatures as down to 625 °C if the Al₂O₃ is deposited on preferably an oxygen rich layer that first is treated with an oxygen containing gas mixture and the subsequent Al₂O₃-process uses high concentration of CO₂ and a sulphur dopant, preferably H₂S. If the oxygen treatment step is excluded then mainly amorphous or metastable phases of Al₂O₃ are formed.

The present invention thus relates to a method of making a cutting tool for metal machining such as turning, milling and

drilling comprising a coating and a substrate. The coating comprises at least one well-crystalline layer consisting of 100 % $\alpha\text{-Al}_2\text{O}_3$ deposited at 625-800 °C using Chemical Vapour Deposition technique. The substrate consists of a hard alloy such as cemented carbide, cermet, ceramics or high speed steel or the superhard materials such as cubic boron nitride or diamond.

The Al₂O₃-layer according to the invention is generally deposited on a substrate that has been precoated with at least one wear resistant inner layer as known in the art. A 0.1-1.5 μm intermediate layer of $\text{TiC}_X N_V O_Z$ where x+y+z>=1 and z>0, preferably z>0.2 is first deposited at 450-600 °C using PVD-technique or at 1000-1050 °C using CVD-technique. Prior to the start of the ${\rm Al}_2{\rm O}_3{\rm -}$ coating step the $\mathrm{TiC}_{\mathbf{X}} \mathrm{N}_{\mathbf{V}} \mathrm{O}_{\mathbf{Z}}$ -layer is treated with a gas mixture containing 0.5-3 vol-% oxygen preferably CO2+H2 or O2+H2, optionally adding 0.5-6 vol-% HCl for a short period of time about 0.5-4 min at temperatures between 625 and 1050 °C, preferably around 1000 °C, if the intermediate layer is deposited by CVD or around 625 °C if the inner layer is deposited by PVD. This step is conducted in order to increase the oxygen content in the surface zone of the intermediate layer. The subsequent Al₂O₃ deposition process is performed with the following concentrations in vol-%: 16-40 CO2, 0.8-2 $\rm H_2S$, 2-10 $\rm AlCl_3$, preferably 2-7 $\rm vol-\$$ $\rm HCl$ and balance $\rm H_2$ at a process pressure of 40-300 mbar and a temperature of 625-800, preferably 625-700, most preferably 650-695 °C.

15

20

25

30

In an alternative embodiment the ${\rm TiC_XN_yO_Z}$ intermediate layer is excluded and the surface of the inner layer is scratched prior to the ${\rm Al_2O_3}$ coating step(s) with hard particles e g diamond in an ultra sonic bath or by a blasting treatment. This applies in particular to a PVD-precoated surface or when depositing at temperatures below 675 °C.

The invention also relates to a cutting tool comprising a body of sintered cemented carbide, cermet or ceramic or high speed steel or the superhard materials such as cubic boron nitride or diamond with at least on the functioning parts of the surface of the body, a hard and wear resistant coating comprising at least one layer consisting essentially of crystalline α -Al₂O₃ with a thickness of 0.5-10 μ m, with columnar grains with an average grain width of 0.1-1.1 μ m and deposited at a temperature of 625-800 °C. Said coating comprises at least one layer consisting of Ti(C,N) with a thickness of 0.5-10 μ m deposited by the MTCVD technique at a temperature less

than 885 °C and preferably with an intermediate layer of 0.5-1.5 μm of $\mathrm{TiC_{x}N_{v}O_{z}}$ preferably x=z=0.5 and y=0 between the α -Al₂O₃-layer and the MTCVD coated Ti(C,N)-layer. Alternatively said coating comprises layer(s) adjacent to the tool body deposited by PVD or 5 PACVD preferably with an intermediate layer of 0.1-1.5 μm TiC_XN_VO_Z preferably with x<0.1 between the α -Al₂O₃ and the PVD- or PACVDlayer(s). In this case the α -Al₂O₃-layer has a pronounced columnar grain structure with a grain width of $<0.5~\mu m$. Preferably one such α -Al₂O₃ layer is the top visible layer at least along the cutting edge line. The coating on the rake face and along the edge line is smoothed by brushing or by blasting to a surface roughness (R_a) of less than 0.2 μm over a measured length of 5 $\mu m.$

The tool coated according to the present invention is a cutting insert or a solid carbide drill or carbide end-mill.

If the grain size of the Al₂O₃-layer is to be determined from a top view projection after the smoothing operation then the Al_2O_3 layer is preferably first etched with a mixture of HF and HNO3 or the grain size can be measured on a fractured sample in a Scanning Electron Microscope as the width of the grains.

The coatings deposited in the examples below were carried out in CVD- and PVD-tool coaters capable in housing several thousands of cutting tool inserts.

Example 1

10

15

20

25

A) Cemented carbide cutting inserts in style CNMG 120408-PM with the composition 7.5 weight-% Co, 1.8 % wt% TiC, 0.5 wt% TiN, 3 wt% TaC, 0.4 wt% NbC and balance WC were coated with a 1 µm thick layer of TiN using conventional CVD-technique at 930 °C followed by a 5 μm TiCN layer employing the MTCVD-technique using TiCl₄, H₂, N₂ and CH₃CN as process gases at a temperature of 700 °C. In subsequent process steps during the same coating cycle, a layer of Ti(C,O) about 0.5 μm thick was deposited at 1000 °C, and then the reactor was flushed with a mixture of 2 % CO2, 5 % HCl and 93 % H2 for 2 min before cooling down in an argon atmosphere to 690 °C at which a 2 μm thick layer of α -Al₂O₃ was deposited according to the invented coating process conditions. The process conditions during the deposition steps were as below:

, PRV 03-04-01 M

Step	TiN	Ti(C,N)	Ti(C,O)	Flush	Al ₂ O ₃
TiCl4	1.5%	1.4%	2 %		
N ₂ CO ₂ :	38 %	38 %		2 %	20%
CO			6 %		3.2%
H ₂ S	-				1 %
HC1	balance	balance	balance	5 % balance	3.2% balance
CH ₃ CN					
Pressure: Temperature: Duration:	160 mbar 930°C 30 min	60 mbar 700°C 4 h	60 mbar 1000°C 20 min	60 mbar 1000°C 2 min	70 mbar 690°C 5 h
	TiCl ₄ N ₂ CO ₂ : CO AlCl ₃ : H ₂ S HCl H ₂ : CH ₃ CN Pressure: Temperature:	TiCl ₄ 1.5% N_2 38% CO_2 : CO $AlCl_3$: H_2S - HCl H_2 : $balance$ CH_3CN - 0.6 $Pressure$: $160 mbar$ $Temperature$: $930^{\circ}C$	TiCl ₄ 1.5% 1.4% N ₂ 38% 38% CO ₂ : CO AlCl ₃ : H ₂ S - HCl H ₂ : balance balance CH ₃ CN - 0.6% Pressure: 160 mbar 60 mbar Temperature: 930°C 700°C	TiCl ₄ 1.5% 1.4% 2 % N ₂ 38 % 38 % CO ₂ : CO 6 % AlCl ₃ : H ₂ S - HCl H ₂ : balance balance balance CH ₃ CN - 0.6 % Pressure: 160 mbar 60 mbar Temperature: 930°C 700°C 1000°C	TiCl4 1.5% 1.4% 2 % N2 38 % 38 % CO2:

15

XRD-analysis of the deposited Al $_2$ O $_3$ layer showed that it consisted only of the α -phase, fig 4. No diffraction peaks from κ -or γ -phase were hence detected.

SEM-micrograph in top-view projection is shown in fig 2. The α -Al₂O₃ layer was astonishingly well crystalline to have been deposited at such low temperature as 690 °C. A grain size of about 1 μ m was observed.

B) Cemented carbide cutting inserts in style CNMG 120408-PM with the composition 7.5 weight-% Co, 1.8 % wt% TiC, 0.5 wt% TiN, 3 wt% TaC, 0.4 wt% NbC and balance WC were coated with a 1 μm thick layer of TiN using conventional CVD-technique at 930 °C followed by a 5 μm TiCN layer employing the MTCVD-technique using TiCl₄, H₂, N₂ and CH₃CN as process gases at a temperature of 700 °C. In subsequent process steps during the same coating cycle, a 0.5 μm Ti(C,0) was deposited at 1000 °C. Then a 2 μm thick α-Al₂O₃-layer was deposited according to prior art technique similar to what is disclosed in US 5,487,625 at 1010 °C. The process conditions during the Al₂O₃ deposition were as below:

	Step	TiN	Ti(C,N)	Ti(C,O)	Al ₂ O ₃	Al ₂ O ₃
	TiCl4	1.5%	1.4%	2 %		
•	N_2	38 %	38 %			
	CO ₂ :				4 %	4 %
5	CO			6 %		
	AlCl ₃ :				4 %	4 %
	H ₂ S	-			•	0.2 %
	HCl				1 %	4 %
	H ₂ :	balance	balance	balance	balance	Balance
10	CH ₃ CN	- 0.6	ક			
	Pressure:	160 mbar	60 mbar	60 mbar	65 mbar	65 mbar
	Temperature:	930°C	700°C	1000°C	1010°C	1010°C
	Duration:	30 min	4 h	20 min	30 min	110 min

XRD-analysis of the deposited Al_2O_3 layer showed that it consisted only of the α -phase.

Example 2

15

40

- C) Inserts in style CNMG 120408-PM with the composition 7.5 weight-% Co, 1.8 % wt% TiC, 0.5 wt% TiN, 3 wt% TaC, 0.4 wt% NbC and balance WC were coated 2 µm of TiN by PVD (ion plating technique).
- D) Cemented carbide substrate of the same style and composition as in C were coated by PVD (ion plating technique) with 25 4 μm of TiN.
 - E.) TiN-precoated inserts from C) were coated with 2 μm of Al2O3 according to the invented process.
 - The coating was performed according to the process:

step	Flush	$Al_{2}O_{3}$
CO ₂ :	3.4 %	20%
AlCl ₃ :		3.2%
H ₂ S·		1 %
HC1	1.5 %	3.2%
H ₂ :	balance	balance
Pressure:	60 mbar	70 mbar
Temperature:	690°C	690°C
Duration:	3 min	5 h

XRD-analysis of the deposited Al_2O_3 layer showed that it consisted of the α -phase. No diffraction peaks from κ - or γ -phase could be detected. A SEM-micrograph in top-view projection of the

obtained coating is shown in fig 1. An average grain size of about 0.25 μm was found.

F.) TiN-precoated inserts from C) were coated with 2 μm of Al₂O₃ according to a prior art process similar to what is disclosed in US 5,487,625.

The coated was performed according to the process:

	step	Al ₂ O ₃	$A1_{2}O_{3}$
	CO ₂ :	4 %	4 %
15	AlCl ₃	4 %	4 %
	н ₂ s		0.2 %
	HCl	1 %	4 %%
	H ₂ :	balance	balance
	Pressure:	65 mbar	65 mbar
20	Temperature:	1010 °C	1010 °C
	Duration:	30 min	110 min

25

XRD-analysis of the deposited Al_2O_3 layer showed that it consisted of the $\alpha\text{-phase}$.

The inserts from A), B), E) and F) were brushed with a nylon brush containing SiC grains in order to smooth the coating surfaces. The PVD coated inserts from D) showed high smoothness already as coated and were therefore not subjected to brushing.

Coating insert from A) and B) were then tested with respect to toughness in a specially designed workpiece. The workpiece consisted of two flat steel plates in material SS1312 clamped together side to side with a distance bar in between leaving a gap between the plates. The plates were cut longitudinal with an increased feed rate until the cutting edge broke. The time to breakage was recorded for each tested insert. Within each variant population some edges last longer than others and the life time of each tested edge was recorded. The obtained result are below presented as time for the insert with shortest life time, time for

the insert with the longest life time and time to when 50 % of the edges within the population had obtained breakage. Ten inserts from A and B were run to edge breakage.

5 Cutting operation 1:

Dry condition

V = 100 m/min

A = 1.5 mm

10 Feed = 0.15-0.35 mm/r

Feed rate increase 0.1 mm/min

Result:

	Time to first breakage, s	Time when 50 % of the insert had failed, s	Time when last insert broke, s
B) Prior art	24	66	83
A) Invention'	62	80	105

Cutting operation 2:

15

A facing operation in an alloyed steel (AISI 1518,W-no 1.0580) was performed. The shape of the work-piece was such that the cutting edge was out of cut three times per revolution.

20 Cutting data:

Speed: 130-220 m/min Feed: 0.2 mm/rev.

Depth of cut: 2.0 mm

Five inserts (edges) were run one cut over the work-piece. The results in table 2 are expressed as percentage of the edge-line in cut that obtained flaking of the coating.

Table 2 Cutting operation 2

Variant	Edge line	
	Flaking average	
B) Prior art	< 10 % only small dots of flaking	
A) Invention	<10 % only small dots of flaking	

From the results from cutting tests 1 and 2 it can be concluded that the inserts according to present invention posses a



30

1

higher toughness and equal flaking resistance compared to prior art inserts.

Cutting operation 3:

5

Cutting inserts from D), E) and F) were tested in a longitudinal turning operation in a ball bearing steel Ovako 825B.

Cutting data:

Cutting speed 210 m/min,

10 Feed 0.25 mm/rev,

Depth of cut 2,0 mm, coolant was used.

The cutting operation was periodically interrupted in order to follow closely the development of the crater wear. The wear was measured (observed) in a microscope. The machining time until the coating broke through and the carbide substrate became visible in the bottom of the crater wear.

Variant	Time to carbide visible
D) PVD TiN precoated	less than 1 min
E) PVD-TiN+ α -Al ₂ O ₃ acc. to the invention	About 5 min
F) PVD-TiN+α-Al ₂ O ₃ acc. to prior art	About 5 min

Cutting operation 4:

20

15

Cutting inserts from D.), E.) and F.) were tested with respect of edge line flaking in a facing operation in an alloyed steel (AISI 1518, W-no. 1,0580). The shape of the work piece was such that the cutting edge was out of cut three times during each revolution.

Cutting data :

Cutting speed 130-220 m/min,

Feed 0.2 mm/rev,

Depth of cut 2.0 mm.

The inserts were run one cut over the work piece. The results are expressed as percentage of the edge line in cut that had obtained flaking.

Variant	Percentage of edge line that obtained flaking
D) PVD-TiN	About 5 %
E) PVD-TiN+α-Al ₂ O ₃ acc. to	About 15 %
invention	
F) PVD-TiN+ α -Al ₂ O ₃ acc. to prior	About 75 % + flaking spread onto
art	the rake face

From the results obtained in cutting operation 3 and 4 it can be concluded that the insert according to the invention has improved crater wear properties over PVD coated tool and better 5 coating adhesion than variant F with a prior art high temperature CVD ${\rm Al}_2{\rm O}_3$ on top of a PVD-TiN layer. Obviously the PVD TiN-pre coating can not withstand the high temperature of the prior art Al₂O₃-process.

Claims

10

15

20

25

1. Method of depositing a crystalline α -Al₂O₃-layer onto a cutting tool insert by Chemical Vapour Deposition c h a r a c t e r i s e d in comprising the following steps depositing a 0.1-1.5 μ m layer of TiC_XN_yO_Z where x+y+z>=1 and z>0, preferably z>0.2

treating said layer at 625-1000 °C in a gas mixture containing 0.5-3 vol% O_2 , preferably CO_2 + H_2 or O_2 + H_2 , for a short period of time about 0.5-4 min, optionally in the presence of 0.5-6 vol% HCl and

depositing said Al_2O_3 -layer by bringing said treated layer into contact with a gas mixture containing 2-10 vol-% of $AlCl_3$, 16-40 vol-% of CO_2 , in H_2 and 0.8-2 vol-% of a sulphurcontaining agent, preferably H_2S , at a process pressure of 40-300 mbar and a temperature of 625-800, preferably 625-700, most preferably 650-695 °C.

- 2. Cutting tool comprising a body of sintered cemented carbide, cermet or ceramic or high speed steel or the superhard materials such as cubic boron nitride or diamond and with at least on the functioning parts of the surface of the body, a hard and wear resistant coating comprising at least one layer consisting essentially of crystalline $\alpha\text{-Al}_20_3$ with a thickness of 0.5-10 μm c h a r a c t e r i z e d that said crystalline $\alpha\text{-Al}_20_3$ has columnar grains with an average grain width of 0.1-1.1 μm and being deposited by Chemical Vapour Deposition at a temperature of 625-800 °C.
- 3. A cutting tool according to claim 2 c h a r a c t e r i z e d in said coating comprising at least one layer consisting of Ti(C,N) with a thickness of 0.5-10 μ m deposited between the body and said α -Al₂O₃-layer by the MTCVD technique at a temperature less than 885 °C.
- 4. A cutting tool according to claim 3 c h a r a c t e r i z e d in said coating comprising an intermediate layer of 0.5-1.5 μ m of TiC_xN_yO_z where x+y+z>=1 and z>0, preferably z>0.2, most preferably z>0.2,y=0 and x>=0, between the α -Al₂O₃-layer and the MTCVD-TiCN-layer.
- 5. A cutting tool according to claim 2 c h a r a c t e r i z e d in said coating comprising at least one layer adjacent to the tool body deposited by PVD or PACVD.

- 6. A cutting tool according to claim 5 c h a r a c t e r i z e d in said coating comprising an intermediate layer of 0.1-1.5 μ m TiC $_X$ N $_Y$ O $_Z$ between the α -Al $_2$ O $_3$ and the PVD or PACVD-layer(s,) where x+y+z>=1 and z>0, preferably z>0.2, most preferably z>0.2, y>=0 and x<0.1.
- 7. A cutting tool according to claims 5 or 6 c h a r a c t e r i z e d in said coating having a pronounced columnar grain structure with a grain width of <0.5 μ m.
- 8. A cutting tool according to any claims 2-7 to a racterized in that one such α -Al₂O₃ layer is the top visible layer at least along the cutting edge line.
- 9. A cutting tool according to any of claims 2-7 c h a r a c t e r i z e d in that the coating on the rake face and along the edge line has been smoothed by brushing or by blasting to 15 a surface roughness, R_a of less than 0.2 μ m over a measured length of 5 μ m.
 - 10. A cutting tool according to any of claims 2-9 c h a r a c t e r i z e d in that said tool is a cutting insert, a solid carbide drill or carbide end-mill.

SUMMARY

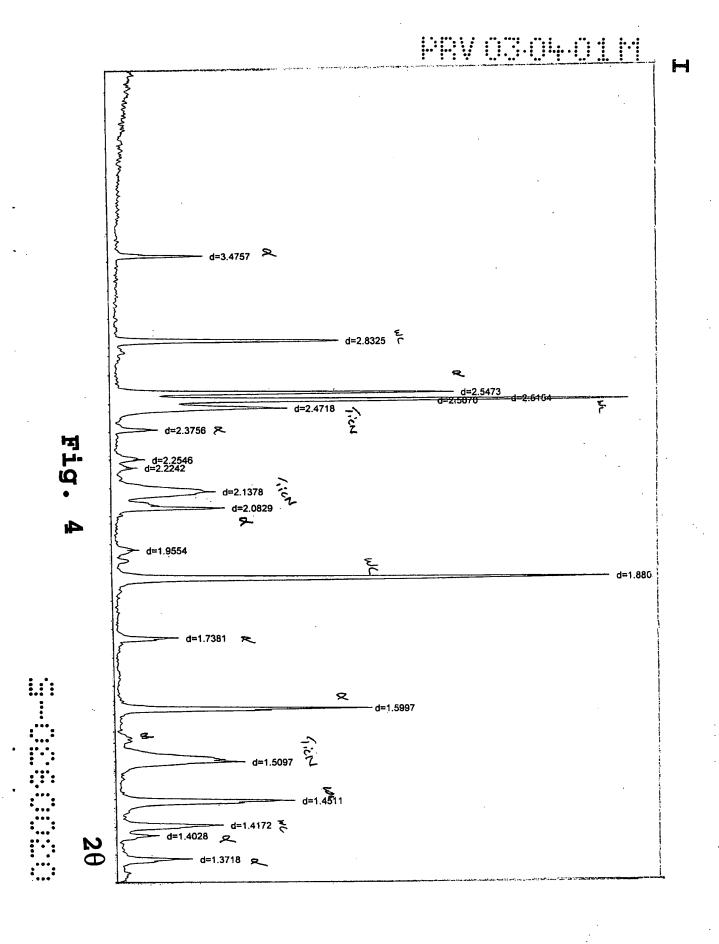
5

The present invention relates to a method of depositing a crystalline α -Al₂O₃-layer onto a cutting tool insert by Chemical Vapour Deposition at a temperature of 625-800 °C. The method comprises the following steps

depositing a 0.1-1.5 μm layer of $\text{TiC}_X N_y O_Z$ where x+y+z>=1 and z>0, preferably z>0.2

treating said layer at 625-1000 °C in a gas mixture containing 0.5-3 vol% O_2 , preferably CO_2 + H_2 or O_2 + H_2 , for a short period of time about 0.5-4 min, optionally in the presence of 0.5-6 vol% HCl and

depositing said Al $_2$ O $_3$ -layer by bringing said treated layer into contact with a gas mixture containing 2-10 vol-% of AlCl $_3$, 16-40 vol-% of CO $_2$, in H $_2$ and 0.8-2 vol-% of a sulphurcontaining agent, preferably H $_2$ S, at a process pressure of 40-300 mbar. The invention also includes a cutting tool insert with a coating including at least one α -Al $_2$ O $_3$ -layer according to the invention.





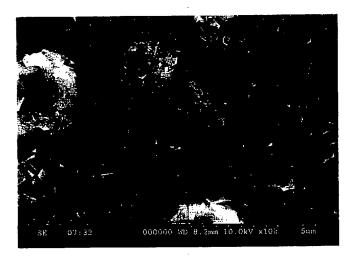


Fig. 1



Fig. 2

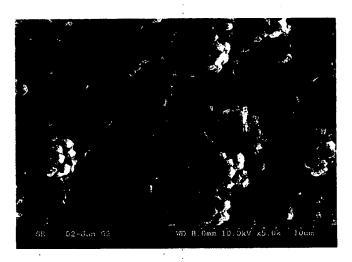


Fig. 3